PART—I

INTRODUCTION

Chapter 1

A Brief review on Hydrazones as analytical reagents

Hydrazones are azomethines containing the characteristic >C=N−N< Group. They are different from imines and oximes etc. because of the presence of two interlinked nitrogen atoms. Metal complexation mainly depends on the selection and sensitivity of the reagents. Organic reagents are treated as special class in the field of complexation of metal ions. These reagents form stable, highly coloured soluble or insoluble complexes with metal ions. These metal complexes find use in analytical chemistry of metal ions. Many organic reagents are available for the determination of most of the elements in the periodic table. Among the numerous organic analytical reagents employed the azomethines have been extensively used to develop methods for their spectrophotometric determinations.

Chapter 2

Medicinal importance of Isonicotinoyl Hydrazone

Isonicotinoyl hydrazones are good analytical reagents due to their ability to form insoluble complexes and their ability to produce characteristics absorption spectra when reacted with certain metal ions. Thus they serve as good reagents even at sub micro gram level. Isonicotinoyl Hydrazones play an important role in medicinal chemistry. Pyridoxal Isonicotinoyl Hydrazone is useful in the treatment of hymphoma and bladder cancer. 3,5-dichloro salicylaldehyde Isonicotinic Hydrazone (SH-7) an analogue of the antituberculosis drug Isoniazid (INH) could prevent Isoniazid-indused liver damage.

Chapter-3

Objectives of the present Investigations

In this chapter the author deals with the objectives of his work, They are

1. To develop simple, rapid, selective, and sensitive methods for the determination of the metal ions in trace quantities

2. To develop sensitive and selective methods for simultaneous determination of metal ions using derivative spectrophotomerty.

Part—II

EXPERIMENTAL

Chapter- 4

Is divided into three Sections

Section-I: Solutions and reagents employed in these investigations.

a. Preparation of Diacetylmonoxime amino isonicotinoyl Hydrazone.

b. Preparation of Diacetylmonoxime isonicotinoyl Hydrazone.

c. Preparation of other inorganic salt solutions.

d. Preparation of buffer solutions.

Section-II: instruments employed- UV-Visible Spectrophotometer and pH meter.

Section-III: General Experimental Procedure Zero order spectrophotomerty

PART-III

Results and discussion

Chapter-5

Studies on Hg(II)- Diacetylmonoxime Aminoisonicotinoyl Hydrazone System

Mercury (II) reacts with DAMAINH to form a yellow coloured complex. Hg(II) forms colour in entire pH range 3-7. The author has chosen pH 6 and

wavelength (λmax) 404.0 nm for further study. At pH 6 the author has studied the effect of metal ion concentration, the effect of reagent concentration, the effect of time, the effect of foreign ions. The stability constants and composition of complex is determined by Job's method and Mole ratio method.

Chapter-6

Studies on V(V)- Diacetylmonoxime amino isonicotinoyl Hydrazone System

Vanadium(V) reacts with Diacetylmonoxime aminoisonicotinoyl Hydrazone to form a greenish yellow coloured complex. The pH of the solution for complete colour formation is found to be 7 and wave length (λmax) is 410 nm. The author has studied the effect of metal ion concentration, effect of reagent concentration, effect of organic solvent, effect of time on the absorbance. Job's method and Mole ratio method are conducted to determine the composition of the complex, stability constant of the complex. The author has studied the interference of various metal ions as well as anions on the absorbance of V(V)-DAMAINH system.

Chapter-7

Studies on Ru(III)- Diacetylmonoxime Aminoisonicotinoyl Hydrazone System

Ruthenium(III) reacts with DAMAINH solution to form a yellow coloured complex. Ru(III) forms colour in entire pH range 5-11. The author has chosen pH 10 and wavelength (λmax) 390 nm for further study. At pH 10 the author has studied the effect of metal ion concentration, the effect of reagent concentration, the effect of time, the effect of foreign ions. The stability constants and composition of complex is determined by Job's method and Mole ratio method.

Chapter-8

Studies on Ni(II)- Diacetylmonoxime aminoisonicotinoyl Hydrazone System

Nickel(II) reacts with DAMAINH solution to form aGolden yellow coloured complex. In a solution of pH 9 complex is showing maximum absorbance hence the author has chosen pH 9 and wavelength (λmax) 385 nm for further study. At pH 9 the author has studied the effect of metal ion concentration, the effect of reagent concentration, the effect of time, the effect of foreign ions. The stability constants and composition of complex is determined by Job's method and Mole ratio method.

Chapter-9

Studies on Ga(III)- Diacetylmonoxime Isonicotinoyl Hydrazone System

Gallium (III) reacts with DAMINH solution to form a Yellow coloured complex. A solution of pH 6 and wavelength (λ_{max}) 362 nm is found appropriate for maximum colour development. The effect of time, effect of organic solvent, effect of metal ion concentration, effect of reagent concentration, effect of foreign ions and their tolerance limits are studied by the author. The author determined the stability constants and composition of the complex employing job's method and mole ratio method.

Chapter10

Studies on Al(III)- Diacetylmonoxime Isonicotinoyl Hydrazone System

Aluminium (III) reacts with Diacetylmonoxime Isonicotinoyl Hydrazone to form a yellow coloured complex. Al(III) forms colour in entire pH range 5-10. The author has chosen pH 8 and wavelength (λ_{max}) 355 nm for further study. At pH 8 the author has studied the effect of metal ion concentration, the effect of reagent concentration, the effect of time, the effect of foreign ions. The stability constants and composition of complex is determined by Job's method and Mole ratio method.

Chapter-11

Studies on Fe (II) - Diacetylmonoxime Isonicotinoyl Hydrazone System

Ferrous iron reacts with Diacetylmonoxime Isonicotinoyl Hydrazone to form a yellow coloured complex. The pH of the solution for the complete colour formation is found to be 9 and wave length (λ_{max}) is 355 nm for further studies. The author has studied the effect of metal ion concentration, effect of reagent concentration, effect of organic solvent, effect of time on the absorbance. Job's method and Mole ratio method are studied to determine the composition of the complex and stability constant of the complex. The author has studied the effect of various metal ions as well as anions on the absorbance of Fe (II) - Diacetylmonoxime Isonicotinoyl Hydrazone System.

PART-IV

Simultaneous determination of metal ions

Chapter-12

Simultaneous determination of Ni (II) and Al (III) using DAMINH

Second order spectrophotometric method is developed for the simultaneous determination of Ni (II) and Al (III) using DAMINH. Two peaks and valleys are noticed for this method. The peak values are 370 nm and 386.0 nm for Ni (II) and Al (III) respectively and valleys are at 375 nm and 396.0 nm for Ni (II) and Al (III) respectively. Ni (II) and Al (III) can be determined in the range 3.98 to 23.8 µg/ml and 0.4692- 2.802 µg/ml respectively.

Chapter-13

Simultaneous determination of Ga (III) and Al (III) using DAMINH

Second order spectrophotometric method is developed for the determination of Al (III) and Ga (III) using Diacetylmonoxime Isonicotinoyl Hydrazone. The peak values are 367.0 nm and 377.0 nm for Al (III) and Ga (III) respectively and valleys are at 372.0 nm and 401 nm for Al (III) and Ga (III) respectively. Al(III) and Ga (III) can be determined in the range 0.499 to 3.049 µg/ml and 3.992- 23.9 µg/ml respectively using this method.

Chapter-14

Simultaneous determination of Fe (II) and Ni (II) using DAMINH

Second order spectrophotometric method is developed for the determination of Fe(II) and Ni(II) using Diacetylmonoxime Isonicotinoyl Hydrazone. The author has recorded individual second order spectra for Fe(II) and Ni(II). The peak values are determined in each case. The peak at 370 nm and valley at 377.0 nm correspond to Fe(II) Similarly the peak at 387.0 nm and valley at 402.0 nm correspond to Ni(II). Fe(II) and Ni(II) can be determined in the range 0.6992 - 6 µg/ml and 0.782 - 7.3 µg/ml respectively.

Chapter-15

Simultaneous determination of Al (III) and Fe (II) using DAMINH

Simultaneous determination of Al(III) and Fe(II) is conducted using second order derivative spectrophotmetry.. The peak at 368.0 nm and valley at 372.0 nm correspond to Al(III) and the peak at 379.0 nm and valley at 392.0 nm correspond to Fe(II). Al(III) and Fe(II) are determined in the range 0.469 - 2.804 µg/ml and 3.98 – 23.8 µg/ml respectively using this method.

Chapter-16

Conclusions

The work presented in the thesis is summarized in this chapter.